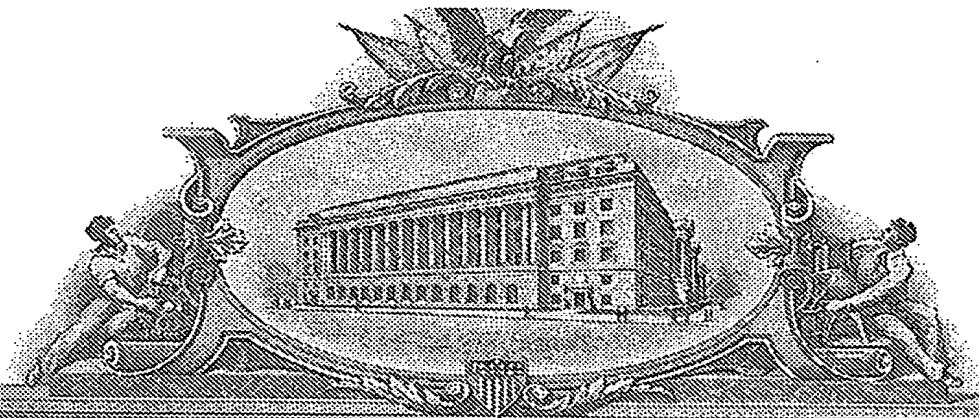


1227869



THE UNITED STATES OF AMERICA

TO ALL TO WHOM THESE PRESENTS SHALL COME:

UNITED STATES DEPARTMENT OF COMMERCE

United States Patent and Trademark Office

January 28, 2005

THIS IS TO CERTIFY THAT ANNEXED HERETO IS A TRUE COPY FROM THE RECORDS OF THE UNITED STATES PATENT AND TRADEMARK OFFICE OF THOSE PAPERS OF THE BELOW IDENTIFIED PATENT APPLICATION THAT MET THE REQUIREMENTS TO BE GRANTED A FILING DATE.

APPLICATION NUMBER: 60/497,527

FILING DATE: August 25, 2003

RELATED PCT APPLICATION NUMBER: PCT/US04/27593



Certified by

Under Secretary of Commerce
for Intellectual Property
and Director of the United States
Patent and Trademark Office

Claims

1. A stable, aqueous froth comprising a) one or more copolymers or interpolymers of ethylene and/or propylene with or without other monomers selected from C₄ to C₁₀ olefins and has an ethylene or propylene content from about 2-98 weight percent; b) water; c) a frothing surfactant; and d) a gas; where the components comprise about: a) 35 to 75 percent, b) 35 to 75 percent and c) 1 to 6 percent of the combined weight of a), b) and c), and d) is present in an amount such that d) comprises at least 80 percent of the total volume of all components present in the froth.
2. The froth of Claim 1 which further comprises component e) a foam stabilizer selected from: cellulose ethers, hydroxyalkyl cellulose ethers, guar gum, xanthan gum, and polyoxyethylene resins of at least 5,000 molecular weight, said component e) present in the amount of from about 0.05 to about 2 percent based on the dry weight of component a) polymer.
3. The froth of Claim 1 where component a) is a copolymer of ethylene with an alpha-olefin comonomer of from 3 to 10 carbon atoms.
4. The froth of Claim 3 where the alpha-olefin comonomer is selected from 1-propene, 1-butene, 1-hexene and 1-octene and component a) has a melt index between about 0.5 and about 30 dg/min under the conditions of ASTM D-1238.
5. A durable, open-cell foam comprising a) one or more copolymers or interpolymers of ethylene and/or 1-propylene, with or without other monomers selected from C₄ to C₁₀ olefins, and which has an ethylene or 1-propylene content from about 2-98 weight percent, wherein the cell size of the majority of cells of the foam ranges between about 5 and about 1000 micrometers (microns).
6. The foam of Claim 5 where component a) polymer of said foam exhibits a melt index between about 0.5 and about 30 dg/min under the conditions of ASTM D-1238.

7. The foam of Claim 5 where component a) comprises at least one copolymer of ethylene with an alpha-olefin comonomer of from 3 to 8 carbon atoms.
8. The foam of Claim 5 where a) comprises an ethylene copolymer with 1-propene, 1-butene, 1-hexene or 1-octene, and which copolymer exhibits a melt index of between about 0.5 and about 30 dg/min.
9. The foam of Claim 5 or 6 where a) comprises an ethylene copolymer with 1-octene, and which copolymer exhibits a density of between 0.85 and 0.91 g/cc.
10. The foam of Claim 7 where the cell size of the majority of cells at a first major surface of the foam ranges between about 20 and about 100 microns and the cell size of the majority of cells at the other major surface of the foam ranges between about 200 and about 1100 microns.
11. The foam of Claim 10 where the cell size of the majority of cells at the first major surface of the foam ranges between about 30 and about 80 microns and the cell size of the majority of cells at the other major surface of the foam ranges between about 300 and about 1000 microns and a gradient in cell size exists between the two said major surfaces of the foam.
12. The foam of Claim 7 where the ethylene copolymer is a copolymer of ethylene and 1-propene and ethylene comprises 2 to 35 weight percent of the copolymer.
13. The foam of Claim 5 where a) comprises an ethylene copolymer with 1-octene, the copolymer exhibits a melt index of between 1 and 20 dg/min under the conditions of ASTM D-1238, a density of between 0.85 and 0.89 g/cc and a cell size gradient of the major portion of cells between one of the major surfaces of the foam and the other major surface exists and ranges between about 20 and about 1100 microns.
14. The Foam of Claim 5 which further comprises at least one additive selected from calcium carbonate powder, titanium dioxide powder, polymer particles, hollow glass spheres, cellulose fibers and film-forming polymers .

15. The Foam of Claim 5 which further comprises one or more of an antioxidant, hydroxyalkylcellulose ether, styrenic latex, guar gum or xanthan gum.
16. An absorbent article selected from a baby diaper, a feminine hygiene product, an adult incontinence hygiene product, a wound dressing pad, a surgical sponge, a food packaging pad, a wiping towel and a wiping sponge, where the article comprises a component made from the foam of Claim 5 or 13.

Froths and Durable Foams of Dispersed Olefin Polymers and

Articles Prepared from Same

Background

Hydrophilic open-cell foams find utility in products for acquiring and distributing aqueous fluids; for example: diapers, adult incontinence pads and briefs, feminine hygiene products, wiping towels and sponges, wound dressings and surgical sponges, and other analogous water-absorption uses. Additionally, both hydrophobic and hydrophilic open-cell foams may find use in numerous other applications, for example fluid filtration, insulation applications, e.g., sound absorption or sound deadening and heat or cold insulation or barriers, cushioning, carpet and fabric backing.

The invention pertains to mechanically frothed compositions comprising aqueous-based, dispersed olefin polymers; recyclable, durable, open-cell foam compositions derived from same; methods for preparation of such froths and foams; and use of the dry durable foams in the absorption, filtration, insulation, cushioning and backing applications by virtue of, among other properties, their good absorption capabilities, softness and/or flexibility and their recyclable nature.

Mechanically frothed derived foam useful in articles for acquiring and distributing aqueous fluids have been prepared from polymeric latex; for example the carboxylated styrene-butadiene latex-derived foams described in USP 4,990,541 and WO-01/80916A2.

Aqueous dispersions of linear olefin copolymers, useful for preparing doctored film coatings are made, but apparently not frothed, using a stabilizing and emulsifying amount of surfactant as described in USP 5,798,410.

Summary of the Invention

The invention comprises aqueous-based, dispersed olefin polymer froths; open-cell, durable foam compositions (dry) derived from same; methods for preparation of such dispersed olefin polymer froths and durable foams; and the use of the durable foams in various applications. More specifically, the relative softness and flexibility of the foams and their good aqueous fluid absorption and aqueous fluid-wicking capability makes them of particular usefulness in absorbent hygiene articles having, among other properties, fluid absorption and wicking capabilities.

We have discovered an open-cell foam that is prepared from aqueous-based dispersed olefin polymer froth and exhibits good Absorbency Capacity ("AC") (expressed as g of synthetic 0.9 wt. percent saline solution absorbed per g dry foam) of greater than 10g/g, preferably greater than 15g/g.

The durable foam of the invention preferably is hydrophilic and for selected applications is capable of vertically wicking the described 0.9% saline solution to a height of greater than about 2 in (~5 cm), preferably greater than about 4 in (~10 cm), and more preferably to greater than about 6 in (~15 cm). Vertical wicking

ability is measured a test of Vertical Wicking Height ("VWH"), described in greater detail below.

The aqueous-based, dispersed olefin polymer froth is prepared from a semi-crystalline thermoformable polymer (Polymer) which provides an added advantage of imparting a recyclable character to the resulting durable foam itself and to articles that incorporate it. In the case of sound and thermal insulation and cushioning applications, in particular, this makes it a very attractive material from which to fabricate articles, e.g., automobile seat cushioning, headliners and sound insulation components, carpet backing for autos or homes, furniture cushioning and mattresses and padding, gas or liquid filtration devices and similar applications where it is highly desirable to have an open-cell foam element that can be simply recycled by heating the article containing the foam to a temperature above the melting range of, and then extracting, the Polymer component of that foam.

The Polymer is selected from copolymers and interpolymers of ethylene and/or propylene and other monomers selected from C₄ to C₁₀ olefins, preferably alpha-olefins, and more preferably selected from n-butene, n-hexene and n-octene. The ethylene or propylene content of Polymer ranges from about 2-98 percent of Polymer. The modulus of the durable foam can be controlled by selection of polymers. For example, using a copolymer having a higher level of C₄ - C₈ olefins will give a softer and more flexible foam than a copolymer having lower amounts of C₄ - C₈ olefins. Similarly, a foam made with propylene/C₄ - C₈ olefin copolymer will give a stiffer foam than a corresponding composition made with ethylene/C₄ - C₈ olefin copolymer. Selected comonomer(s) make up the remainder of the Polymer. Further details regarding the Polymer are found below.

The absorbent, open-cell Foam composition of the invention is a durable Foam that results from drying of the aqueous, frothed dispersion of Polymer under conditions selected to inhibit the coalescence of the individual gas bubbles in the Froth for a time period sufficient to allow dispersed Polymer particles contained in the thin aqueous layer surrounding the entrapped air bubble to fuse before the ' aqueous film structure undergoes significant collapse, as the water evaporates from the bubbles' surfaces and from the channels or interstices between the bubbles.

Some preferred modes of use and articles comprised of the absorbent Foam include aqueous-fluid absorbent, conformable hygiene articles, more particularly baby diapers, adult incontinence products, feminine hygiene products, nursing pads, sweat bands, wiping toweling and sponges, wound dressing pads, surgical sponges, and food packaging absorbent padding such as is employed for absorbing meat juice and drippings at the bottom of food packaging trays.

The invention further comprises recyclable, absorbent articles. In the case of generally non-disposable articles of a more permanent and reusable character, such as sound and thermal insulation and cushioning applications, in particular, the recyclable nature makes the absorbent Foam a very attractive material from which to fabricate articles due to their impact-absorption, sound absorption or other absorption-related properties; for example, automobile seat cushioning, headliners and sound insulation components, carpet backing for autos or homes, furniture

cushioning and mattresses and padding, gas or liquid filtering devices and similar applications.

Detailed Description of the Invention

All percentages and parts, unless otherwise stated, are expressed by weight.

Definition of Terms

The term Conformable as used here means the ability to bend and flex to the desired shape of the user; for example, the shape of a wearer of an absorbent article.

The term Dispersion as used here means a two phase liquid/polymer composition where the aqueous phase is the continuous phase and the Polymer is suspended therein in a stable fashion, suitably with the aid of a dispersing agent/dispersant so that the polymer will remain dispersed at least for as long as it will require to complete the frothing step. Preferably the polymer will remain dispersed throughout the entire frothing and drying process so that a complete process can be conducted, either batch-wise or in a continuous fashion, without the polymers settling out of from the dispersion. It is not critical to this invention how the dispersion is obtained. Suitable methods are taught in the art; see for example USP 5,798,410 and USP 6,448,321.

The term Drying as used here means a process of causing a Froth to become a Dry Foam and the term Dry as used herein means elimination of at least 95 percent of the water from the Froth.

The term Frothing or Frothed as used here means a process of incorporating substantial volumes of air or other gas in a liquid, where at least 80, preferably at least 85 and more preferably at least 90 volume percent of the frothed material consists of the gaseous component. It is understood that the aqueous liquid can be a molecular solution, a micellar solution or a dispersion. In general the froth is created by mechanical methods such as high shear mixing under atmospheric conditions or optionally injecting gas into the system while mixing.

The term Froth as used here means an aqueous dispersion of the Polymer which has been Frothed, as described above.

The term Foam as used here means a durable structure having an open cell content of at least 80%, preferably greater than 85% and more preferably at least 90 percent, as determined by and according to ASTM D2856-A. In the invention, a Foam is suitably prepared by removing the liquid/aqueous element of a Froth prepared in the manner of the invention teaching. It is desirable to minimize the amount of froth volume collapse during this conversion. In general foams of this invention will have volume losses of not greater than about 30% during drying.

The term Major Surface as used here means, in a Foam, one of two substantially parallel surfaces of largest area, in contrast to a minor surface thereof. While possible to cut and trim a raw foam in a manner to form a six surface, regular three dimensional cubical geometrical structure, where all 6 Foam surfaces are of substantially the same area, because of the practical nature of continuously generating a foam article, it's normally accomplished by spreading frothed material

on a conveyer moving in the x-direction, of y-dimension Froth width, and z-dimension Froth thickness. Two major Froth surfaces of x-length and y-width, which when dried to Foam results in a three dimensional Foam structure of surface area equal to about xy on both the top and bottom. It is each of these top and bottom Foam surfaces that are referred to here as a Major Surface, or in the case of Foam that has been slit into pieces of approximately equal thickness along the x-y axes, Major Surface means the resulting larger parallel surface on each opposite, parallel side of each of the resulting slit sheets of Foam.

Olefin Polymers

The semi-crystalline olefin polymer (Polymer) is selected from copolymers and interpolymers of ethylene and/or propylene and other monomers selected from C₄ to C₁₀ olefins, preferably alpha-olefins, more preferably from C₄ to C₈ alpha-olefins and most preferably selected from n-butene, n-hexene and n-octene. The ethylene or propylene content of the Polymer ranges from about 2-98 weight percent of Polymer. Where a softer, more flexible foam is desired a primarily ethylene-based polyolefin is selected in which ethylene comprises from about 98 to 65 percent of Polymer. Where a stiffer foam of greater flexural modulus is desired, a primarily propylene-based polyolefin may be selected, propylene comprising from about 98 to 65 percent of the Polymer. Selected comonomer(s) make up the remainder of the Polymer.

The Polymer has the following characteristics and properties:

- 1) Crystallinity as determined by the observance of at least one endotherm when subjected to standard differential scanning calorimetry (DSC) evaluation;
- 2) a melt index ("MI") [determined according to ASTM D1238 at 375 deg F. (190deg C.) with a 4.75 lb (2.16 kg) weight] of between about 30 and about 0.1 dg/min, preferably of between 25 and 0.25 dg/min, more preferably of between 22 and 0.5 dg/min and most preferably of between 18 and 0.75 dg/min; and
- 3) a density of between about 0.845 and about 0.925 g/cc, preferably between 0.85 and 0.91 g/cc, and more preferably between 0.855 and 0.905 g/cc, and most preferably between 0.86 and 0.90 g/cc.

One class of Polymers particularly suited to use in the invention are copolymers of ethylene and 1-octene, where ethylene comprises from about 90 to about 50, more preferably 85 to 55, and 1-octene from about 10 to about 50, more preferably about 15 to 45 percent by weight of the copolymer, and that have a Melt Index of between about 0.25 and about 30 more preferably between 0.5 and 20 dg/min.

Alternatively, instead of Polymer, a blend of polymers may be employed that has the physical characteristics described above. For example, It may be desirable to blend a first polymer with relatively high MI that is outside the range described above, with another of relatively low MI, so that the combined MI and the averaged density of the blend fall within the ranges noted above. A more crystalline alpha-olefin polymer may be combined with one of relatively lower crystallinity, such as one having a significant amount of long chain branching, to provide a blend that has substantially equivalent processing capability in preparing stable Froth and durable

Foam of the invention. Where reference is made to a "Polymer" in this specification, it is understood that blends of olefin polymers with equivalent physical characteristics may be employed with like effect and are considered to fall within our description of the invention.

A particularly preferred class of Polymer when used without other polymers or film forming additives, is characterized by exhibiting a particular type of DSC plot of the endotherm. In the preferred class, the observed endotherm exhibits a relatively gentle positive slope as the scanning temperature is increased past the final endotherm maximum (i.e., the last inflection point on the DSC curve where the curve slope becomes positive and the curve then returns to baseline state). This reflects a polymer of broad melting range rather than a polymer having what is generally considered to be a sharp melting point.

Consequently, the drying temperature of Froth prepared from such a preferred Polymer can be more easily maintained at or near a point on the endotherm curve a significant distance from the return to baseline position at which point a major part, but not all, of the crystalline portions the Polymer are melted and allow the remaining Polymer particles to fuse or coalesce via their amorphous regions. In that fashion, during the drying process by maintaining such a temperature most of the Polymer is allowed to fuse without a complete loss of Polymer tensile strength and the bubble collapse that would otherwise ensue, if all crystalline portions of the Polymer were to be melted quickly.

Dispersing Agents (Dispersants)

The Dispersant is employed in an amount of more than about 1 wt %, preferably more than about 2 wt %, and more preferably more than about 3 wt %; up to an amount less than about 10 %, preferably less than about 8 wt %, and more preferably less than 5 wt. %, based upon the weight of the aqueous dispersion of the Polymer.

The Dispersant used to create the relatively stable, aqueous dispersion of Polymer particles in the invention will vary by the nature of the Polymer employed. The same or a different Dispersant may be used to serve as the frothing surfactant in the subsequent preparation of the Froth.

Suitable Dispersants for the Polymer are salts of fatty acid(s) of carbon chain length of greater than 12 and preferably from 18 to 36 carbon atoms. The salts are suitably alkali metal or ammonium salts of the fatty acid, prepared by neutralization of the acid with the corresponding base, e.g., NaOH, KOH, and NH₄OH. These salts may be formed *in situ* in the dispersion step, as described more fully, below. The appropriate fatty acid Dispersant is selected to serve as Dispersant for the extrusion melt step in order to attain the desired average particle size of the Polymer, suitably between about 0.2-25 microns and preferably between about 0.5-10 microns.

Frothing Surfactants

Creating and stabilizing the Froth during the frothing and drying step is suitably accomplished by addition of a frothing surfactant to the aqueous dispersion of the Polymer when initially creating the Froth. In addition these surfactants can be used to improve aqueous wetting if desired. Suitable frothing surfactants are selected

primarily from but are not limited to, cationic, nonionic and anionic surfactants, with anionic surfactants being preferred.

Cationic surfactants such as primary amine salts, diamine salts, quaternary ammonium salts and ethoxylated amines and the like., Nonionic surfactants such as alkylphenol ethoxylates, linear and secondary alcohol ethoxylates of alkyl group containing more than 8 carbon atoms.

Anionic surfactants to be used in preparation of the Froth from a previously created Dispersion of the Polymer are suitably selected from carboxylic acid salts and ester amides of carboxylic fatty acid, preferably saturated fatty acids comprising from 12-36 carbon atoms, e.g., stearic or lauric acid, and more preferably unsaturated fatty acids such as oleic, linoleic, ricinoleic, erucic acid and the like. More preferred are those of comprising from 12-24 carbon atoms, particularly their alkali metal (most preferably sodium or potassium) or ammonium salts. Other suitable anionic surfactants include alkylbenzene sulfonates, secondary n-alkane sulfonates, alpha-olefin sulfonates, dialkyl diphenylene oxide sulfonates, sulfosuccinate esters, isethionates, linear alkyl (alcohol) sulfates and linear alcohol ether sulfates. It is understood that these frothing may or may not be different than those used to prepare the dispersion. These surfactants serve both to assist in Froth formation and help to stabilize the Froth.

The most preferred frothing surfactants are the alkali metal (more preferably sodium or potassium) and ammonium salts of lauryl sulfate, dodecylbenzene sulfates, alcohol ethoxy sulfates, isethionates, and such dibasic salt of N-octyldecylsulfosuccinimate, as well as mixtures of same.

Dispersion Step

The selected Polymer is dispersed in water, by adding the Polymer and the selected Dispersant(s) in the desired amounts and in a metered fashion to the hopper of a bi-axial, polymer extruder, where they are melt-kneaded at a temperature of about 430 deg. F. (about 220 deg C.). Preferably, when using an ethylene-based olefin Polymer, the long chain fatty acid of greater than 18 carbon atoms is melt-kneaded with the Polymer and then deionized water and base, e.g. KOH sufficient to form *in situ* the fatty acid salt of Dispersant, are added at about 310 deg F. (~165 deg. C.) to the melt under a pressure of at least 410 psi (~2,800 kPa) to produce the Dispersion. Pressure within the extruder barrel is maintained above the saturated steam pressure of roughly 20 to 35 atmospheres (~2,000 to ~3,500 kPa) to avoid "blowback" through space between the barrel and screw of the extruder, by ensuring that space is essentially full of the Dispersion. Then the Dispersion is diluted with deionized water at a separate port downstream in the extruder barrel at about 380 deg F. (~193 deg C.) and at about 14 atmospheres (~1,400 kPa) to produce a final Dispersion of about 67 percent solids. Dispersion is conducted from the extruder, after passing through a mild cooling zone to prevent flashing of the water from the Dispersion, at a temperature of about 200 deg F. (~94 deg C.).

Froth Preparation

A froth is prepared from the aqueous dispersion of the Polymer by using a high shear, mechanical mixing process to entrain air or another gas in the aqueous phase

of the dispersion. The amount of air or other gas (where a gas in addition to or other than air is desirable) to be incorporated in the Froth suitably comprises at least 80, preferably 90 percent by volume of the resultant Froth. In general, all components to be used in making the froth are mixed together with mild agitation to avoid entrapping air. Once all of the ingredients are well mixed, the composition is exposed to high shear mechanical mixing. During this step the bulk viscosity increases as more air is entrapped within the continuous aqueous phase. The mixture is mixed until a non-flowable, stiff froth is formed. This generally produces a froth with density of less than about 100g/l. The time to reach this stage varies with amount and type of frothing surfactant and the amount of mechanical shear. Any mechanical mixing device capable of whipping air into a thickened aqueous dispersion, such as a kitchen blender/hand mixer, Hobart mixer fitted with a wire whip or on a larger scale a Cowie-Riding Twin Foamer (Cowie Riding Ltd., G.B Patent 1,390,180). The commercial foamers also allow one to inject air into their high shear mixing head to obtain very low froth density (less than 50g/l).

Additives

The Foam of the invention may contain filler materials in amounts, depending on the application for which they are designed, ranging from about 2-100 percent (dry basis) of the weight of the Polymer component. These optional ingredients may include, but are not limited to, calcium carbonate, titanium dioxide powder, polymer particles, hollow glass spheres and the like. Foam designed for use in the absorbent articles may contain bulk liquid-absorbing material, such as short cotton fiber or other cellulose fiber evenly distributed throughout the polymer foam. Although they are not blended with the Polymer dispersion before frothing, due to their strong water absorbent nature, fine particles of super absorbent polymer ("SAP") a lightly cross-linked acrylate polymer, can be evenly distributed upon the surface of the Froth just as it is entering the drying process to provide a durable Foam with extra absorbent properties on that surface when dried.

Other Foam end-uses such as cushioning, particularly flooring backing, can benefit from the addition of low cost fillers such as calcium carbonate or titanium dioxide powder, and similar inert fillers. Such additives and fillers can enhance the physical strength and/or the appearance of the resultant composite Foam after drying, as well as retain or to increase the Foam's impact or other absorption capabilities. For example, about 1-25% such cellulose fiber material of fiber length of about 0.2-0.4 in (about 5-20 mm) may be added without substantially affecting the absorption performance or structural integrity of the Foam and in fact do enhance durability and structural integrity of the Foam. For some Foam applications, it may be desirable to incorporate one or more antioxidants and/or other stabilizing agents to enhance the resistance of the Foam to oxidization and yellowing from exposure to harsh conditions and weathering.

Synthetic latex polymers (e.g., styrenic or acrylic lattices) and/or other film-forming polymers may also be utilized as additives to the Froth to form stable and durable Foams and may aid in processing of the Froth and conversion to Foam by contributing to enhanced coalescence of the Polymer particles at both lower and higher drying temperatures. If utilized, such lattices or other film-forming polymers

are suitably employed at levels of about 10-40 percent, dry weight basis, of the Polymer.

When additives are to be incorporated in the Foam, they are suitably be added in the specified amounts to the dispersion of the Polymer before the Froth is prepared in the frothing step. However, as previously noted above, when water soluble or highly hygroscopic additives (such as SAP) are desirable to add, they are added to the Froth surface immediately before the drying step or injected into the finished durable Foam.

Froth Stabilization Agents

Water-soluble, film-forming natural and synthetic polymers such as those selected from cellulose ethers and hydroxyalkyl cellulose ethers e.g. methylcellulose; hydroxypropyl methylcellulose (HPMC); hydroxyethyl methylcellulose (HEMC); hydroxyethyl cellulose (HEC); hydroxypropyl hydroxyethylcellulose HPHEC; polyoxyethylene (water-soluble, high molecule weight polymers of ethylene oxide, preferably of about 5,000 molecular weight or higher, such as POLYOX resins offered by Union Carbide Corp.); natural products such as guar gum, xanthan gum and similar water-soluble thickening agents, will serve as stabilization agents ("Stabilizers") for the frothed Polymer dispersion. From about 0.05 to about 2 percent of stabilizer, preferably about 0.1 to about 1 percent, and most preferably about 0.2 to about 0.5 percent, based on the dry weight of the Polymers.

Treatment Equipment and Process Conditions

The Froth and Foam of the invention may be prepared any suitable equipment normally employed for frothing of aqueous dispersions and drying of such froths. Any mixing or stirring device useful for preparation of aqueous particulate dispersions can be utilized in preparation of the dispersion and subsequent formulation blending with surfactants and other additives, with care being taken to avoid entraining air in the blend before frothing commences. A kitchen blender or other bladed mixing device is suitable. When the blend is prepared, the same or different mixing device can then be operated to commence air entrainment in the formulated aqueous blend containing the Polymer and other additives. A specifically designed frother such as a Cowie-Riding twin foamer may also be used to prepare the Froth, so that the desired target 80-90 or 95% air content of the Froth, depending on the desired density of the final Foam, may be attained. The correct amount of frothing and air content can be easily determined by a few simple experiments. Froth density is measured by drawing off samples of the Froth in cups of predetermined volume and weight, weighing the Froth-filled cup and then calculating the density of the sample. In commercial frothers air can be added directly into the mixing head to assist in development of low density froths. The speed of the frothing device can then be increased or decreased to attain a desired Froth density.

Drying of the Froth to form the desired Foam may be conducted in batch or continuous mode. Devices such as conventional forced air drying ovens or banks of infrared heating lamps lining a tunnel or chamber in which the Froth may be placed or conveyed through, in a continuous fashion, for drying may suitably be employed

for this purpose. The temperature of operation is selected according to the nature and the melting range of the Polymer (as determined by DSC) employed to prepare the Foam, as described immediately below.

Drying and Recovery Steps

The Froths of the invention are dried and converted to invention Foams by heating them in a forced air drying oven, at temperatures selected for optimum. Typically the Froth is heated to a temperature between about 160 and 250 deg F. (~ 70 and ~120 deg C.). As the nature of the Polymer permits, processing at the highest temperature possible to remove water as rapidly as possible from the Froth without destroying the viscosity of the Polymer on the surface of the bubbles of the Froth or causing significant (e.g. more than 30 volume percent) collapse of the partially dried froth. Typically, it is desirable to perform the drying step at a temperature that approaches, but does not exceed the Polymer's melting range. The desired condition is to attain a temperature where the amorphous regions in the Polymer can begin to coalesce while the pseudo-crosslinking in the Polymer created by the crystalline regions in same still are capable of imparting sufficient viscosity to the heated Polymer to avoid collapse of the Froth before the Foam has become fully "dried" in its ultimate form and dimension and at least 95 weight percent of the water in the Froth has been driven out.

The melting range of a Polymer is determined by Differential Scanning Calorimetry (DSC) techniques, and the temperatures bracketing the region of the DSC endotherm, or the final endotherm if more than one exist, just before a return to baseline on the DSC scan plot is the temperature range in which drying of the Froth to form the finished Foam is to be conducted. As described earlier, the particularly preferred Polymers when used without other polymers or additives are characterized by exhibiting a specific DSC plot of the endotherm. In such Polymers, the observed endotherm exhibits a relatively gentle positive slope as the scanning temperature is increased past the final endotherm maximum (i.e., the last inflection point on the DSC curve where the curve slope becomes positive and the curve then returns to baseline state). This reflects a polymer of broad melting range rather than a polymer having what is generally considered to be a sharp melting point.

Consequently, the drying temperature for a Polymer is best maintained at or near a point on the endotherm curve a significant distance from the return to baseline position at which point a major part, but not all, of the crystalline portions the Polymer fuse and Polymer particles fuse/coalesce. In that fashion, during the drying process by maintaining such a temperature most of the Polymer is allowed to fuse without a complete loss of Polymer tensile strength and the bubble collapse that would otherwise ensue, if all crystalline portions of the Polymer were to be melted quickly.

When a blend of Polymer with additives (including blends with other thermoformable polymers) is to be employed in the preparation of invention Froth and Foam, a DSC plot for the blend is first suitably rendered. From that plot endotherm(s) of the blend may be observed and consequently the final melting range of the blend determined and suitable drying temperature for converting the Froth to durable Foam selected.

Foam Slitting Step

While Foams of the invention may be utilized directly as laid and dried, particularly in commercial operations where a sheet of Froth and resultant Foam may be continuously produced, it is frequently desired to form a durable Foam of roughly twice the thickness of the Foam to be used in the finished article. Then to slit the Foam along the axis of the continuous flow into two Foam sheets of about the same thickness.

By virtue of the fact that a foam will normally dry more quickly at its outer, exposed surfaces than in the interior of the foam, the size of the open cells on the outer surfaces when dried will normally be smaller than the size of the cells in the interior. This is a result of the fact that there will be a certain amount of bubble coalescence and resultant bubble diameter enlargement during the drying step. The size of the bubbles/final cell size is a function of time required to dry the Froth and for the Polymer particles to merge to form cell walls in the final Foam. The longer the time, the more bubble coalescence will occur, more bubble diameter enlargement will take place and the larger will be the ultimate cell size in the finished Foam. Accordingly, unless a uniform drying throughout the Foam can be accomplished there will be some cell size gradient from the surface to the interior of the Foam as formed. This result is desirable in many applications such as sound insulation, and in absorbent applications where the foam needs to uptake fluid quickly, yet wick fluid away from one surface of the Foam, e.g. in diaper, adult incontinence articles or feminine hygiene articles, is the ultimate goal.

After slitting of the Foam through its approximate center, the resulting two pieces of Foam will exhibit the cell size gradient from one surface to the other in a mirrored fashion for each half of the slit Foam sheet. In use of the slit Foam for wicking applications, the Foam is oriented so that the larger cells of one major surface will initially contact the aqueous solution to be absorbed and then the capillary action of the increasingly smaller cells traveling toward the other major surface will cause the solution to be wicked away from the first major surface and to be distributed nearer to the second major surface. The result is a dryer surface near the point of initial contact with the Foam, providing the desired effect in such diaper, medical and hygiene applications of the Foam.

For use in absorbent articles, a Foam exhibiting a cell size gradient between the two Major Surfaces is highly desirable, larger cells at one Major surface allowing for a quick uptake of fluid insults and smaller cells at the other Major Surface imparting a wicking action to the Foam element in a final article to move fluid away from the insult site. Suitably, the cell size gradient is such that the major portion of cells fall into a range from about 10 microns at one Major Surface of slit Foam to about 1100 microns at the other Major Surface. The major portion of cells on one Major Surface is normally found in a range of between about 20 to about 100 microns and the major portion of cells at the other Major Surface to be found in the range between about 200 to about 1000 microns; more preferably said gradient varies from cells ranging between about 30 and about 80 microns at one Major Surface and between about 250 and about 800 microns at the other Major Surface, and most

preferably said gradient varies from about 30 microns at one to about 80 microns at the other Major Surface.

Cell size gradient can be influenced by faster or slower drying of the Froth, with slower drying generally resulting in a Foam of wider cell size gradient and faster drying in one of narrower size gradient. Addition of a more effective Froth stabilizer, such as a hydroxyalkyl cellulose ethers, or film-forming additive such as a styrenic latex, may result in a Foam with a narrower cell size gradient as well.

Foam Cell Size and Measurement/Calculation Methods

The size of cells in the durable Foam is determined by first obtaining an image of the cells using a Scanning Electron Microscope (SEM) to provide a black and white image of surfaces or cross sections of the Foam. The SEM image is then subjected to electronic image scanning and the data from the scan is analyzed by SCION imaging software available from SCION Corp. to provide a cell size plot for a given image area. The data may be graphically displayed for further analysis as a function of distance along some chose axis of the B/W image from which input was collected to visually display the structural nature of the Foam.

Testing Procedures

Unless otherwise indicated, the following test procedures are employed to measure the characteristics/performance of the Foam samples.

Vertical wicking height ("VWH") of Foam. This test is employed to quantify the ability of an open cell to move fluid away from the insult site (i.e., the point of contact by the "insult" fluid to be wicked away). A sample strip of the Foam, approximately 1 inch (2.54 cm) wide and approximately 12 inches (30.5 cm) long is adhered to a plastic plate using double-sided tape and positioned adjacent and parallel, in the longer direction, to a ruler or other similar measurement tool such that the bottom of the strip is positioned with the 0 indicator marker on the ruler. The plate with sample is then suspended over a bath of the 0.9% aqueous saline solution that contains a minimal amount of a food coloring (to assist observation of the movement of the fluid front in the foam strip). At time "zero" the bath is raised to just contact the bottom edge of the Foam strip. The height from the bottom edge of the strip, of the fluid front on the strip surface is recorded at selected time intervals, generally at 2, 10, 30 and 60 minutes.

Absorbent capacity ("AC") is determined using a pre-weighed (dry weight) foam sample. The sample is fully immersed in a bath of the same 0.9% aqueous test solution. Once fully saturated, it's removed from the bath with tweezer or a spatula. It is placed on a coarse wire mesh where excess fluid is permitted to drain for approximately until visible fluid flow from the sample ceases and the saturated Foam sample is weighed to establish the "wet weight". AC is then calculated by dividing the [wet weight – dry weight] difference by dry weight of the strip.

Specific Embodiments of the Invention

All reported percentages are by weight, unless otherwise stated.

Examples 1 through 5 demonstrate the dispersion of the Polymer, frothing of the Polymer dispersion, drying of the Froth to form the durable Foam and the ability of the Foam to absorb and wick saline aqueous solutions (e.g. synthetic urine or synthetic blood samples, or "insults").

Example 1

Dispersion

A dispersion of an ethylene/1-octene copolymer is prepared from a copolymer having ethylene/1-octene content of 62/38 percent (ENGAGE 8200 elastomer which is supplied by DuPont Dow Elastomers), and having a density of 0.870 g/cc and a melt index of 5 dg/min (as determined by ASTM method D1238 at 375 deg F. with a weight of 4.75 lbs).

In the manner described earlier under the heading "Dispersion Step", 10,000 parts of the copolymer is fed into the hopper of the polymer extruder together with 640 parts (active weight) of dispersant (Unicid 350, a dispersant a 26 carbon chain fatty acid as active component) and melt-kneaded by a single screw extruder at about 430 deg F. Thereafter into the barrel of the twin-screw extruder 70 parts potassium hydroxide in 850 parts deionized water are added to the polymer/dispersant blend under pressure and a temperature of about 310 deg F. As the blend passes down the extruder barrel, further deionized water is added until a final dispersion of about 59 % solids is produced. The resulting dispersion is cooled to about 200 deg F. before exiting the extruder and then recovered.

Froth Preparation

A sample of 196.5 parts of the recovered dispersion (51% active or 100 parts solid Polymer) is blended, in a conventional mixing bowl and taking care not to entrain air while blending, with 3.25 parts of a 30% solution (0.98 part active) of the selected frothing surfactant (sodium lauryl sulfate) and with 8 parts (0.33 part active) of a 2.5% aqueous solution of the hydroxyalkyl cellulose ether Methocel E4M hydroxypropyl methylcellulose thickener supplied by The Dow Chemical Company, into 100 parts deionized water.

Small samples are prepared with a KitchenAid Professional 9-speed hand mixer (Model KMH9PWH) and larger samples are prepared with a Hobart-type stand mixer KitchenAid Professional mixer (Model KSM50PWH) and a pair of wire beaters. After the initial blend is prepared, air is entrained by mechanical frothing using the same mixers, but fitted with a wire whip and by increasing the mixer speed from medium to high over a period of approximately 5 to 10 minutes, until a stiff froth is formed. Density of the froth is measured by weighing a 3 oz (89 ml) paper cup filled with foam. Once the desired density is reached, whipping is stopped.

Foam Preparation/Drying

Froth prepared above is spread on release paper supported by a stiffer web sheet and is smoothed to a height of 0.5 in. The froth is placed in a Blue M forced air oven the selected drying temperature of approximately 165 deg F. for about 65 minutes. The foam sheet is recovered and slit lengthwise along the axis that parallels the two major surfaces to yield two mirror image sheets of foam having small cell sizes

ranging from about 30 to 100 microns on their outer surfaces and larger cell size ranging from about 250 to 800 microns on their inner major surfaces.

Example 2

In the manner described in Example 1 above, dispersions, froths and foam samples are prepared. The types and characteristics of the Polymer, froth stabilizer, any additive and surfactants, and the properties of the Foams are described in the following Tables. The Polymer is selected from a broad series of ENGAGE ethylene/1-octene copolymer elastomeric resin, a resin available from DuPont Dow Elastomers. Unacid 350 dispersant is a 26 carbon (average chain length) fatty acid. Unacid 425 dispersant is a 32 carbon (average chain length) fatty acid. The fatty acids are utilized in their potassium salt as formed in the extrusion step described above. Frothing surfactant Stanfax 318 surfactant is sodium sulfosuccinimate and Steol CS-130 is a sodium long chain alkyl ether sulfate. The last two surfactants are added with dilution water near point of exit from extruder in the extrusion step described above.

TABLE - 1
Polymer Composition

<u>Polymer</u> <u>Designation</u>	<u>Ethylene/1-Octene</u> <u>Content</u> (wt %)	<u>Density</u> (g/cc)	<u>Melt Index</u> [@ 190 deg C./2.16kg] (dg/min)
1A	55/45	0.857	1
1B	58/42	0.864	13
1C	60/40	0.870	30
1D	62/38	0.870	5
1E	65/35	0.875	3
1F	67/33	0.880	18
1G	69/31	0.885	30
1H	78/22	0.902	30
1J	80/20	0.902	3

TABLE - 2
Polymer Dispersion Characteristics

<u>Dispersion Designation</u>	<u>Dispersant (wt % based) (on total solids)</u>	<u>Polymer & Content (wt % solids)</u>	<u>Particle Size (microns)</u>	<u>Polymer Melting Temp. Range (deg C.)</u>
2.1	6:% Unicid 425	1A 60.2%	1.56	25-60
2.2	6% Unicid 425	1E 54.5%	1.69	30-90
2.3	6% Unicid 425	1J 53.9%	1.18	65-110
2.4	6% Unicid 350	1B 59.0%	0.55	25-70
2.5	6% Unicid 350	1C 57.2%	0.72	25-80
2.6	2% Unicid 350	1C 54.8%	1.02	ditto
2.7	6% Unicid 350	1F 55.0%	0.69	----
2.8	6% Unicid 350	1G 55.6%	0.71	25-100
2.9	6% Unicid 350	1H 50.6%	0.70	50-110
2.10	6% Unicid 350	1D 50.9%	0.84	30-80
2.11	2% Unicid 350	1D 53.0%	0.95	ditto
2.12	3% erucic acid	1D 48.4%	0.85	ditto
2.13	3% oleic acid	1D 55.6%	2.23	ditto
2.14	2% Unicid 350 + 2% Stanfax 318	1D 55.2%	1.17	ditto
2.15	2% Unicid 350 + 4% Stanfax 318	1D 54.1%	1.05	ditto
2.16	2% Unicid 350 + 2% oleic acid	1D 58.2%	1.56	ditto
2.17	2% Unicid 350 + 2% Steol CS 130	1D 51.8%	1.06	ditto

In Dispersions 2.14, 2.15 and 2.17, the Stanfax and Steol products are added to perform as the Frothing Surfactant in a following frothing step.

Example 3 – Froth and Foam Preparation

Samples 3A, B, C and D of dispersions are prepared in similar fashion as those designated above in Table 2 as Dispersion 2.10 and Dispersion 2.12 from Polymer 1D. Sample 3A uses 6% Unacid 350 dispersant, while Samples 3b, C and D each use 3% erucic acid as dispersant in preparation of the base dispersion. Each of the dispersion Samples 3A-D is frothed with 1% Steol CS-130 surfactant, together with 0.2% active weight of Methocel E4M hydroxypropyl methylcellulose in the manner described earlier. The froths are dried at approximately 167 deg F. (75 deg C.) in a forced air oven. Sample 3D is dried in a slightly different fashion, being placed in an infrared heated "oven" and passed through quickly first, before being placed in the standard forced air oven to complete the drying process. This serves to quickly dry the foam surface than the other technique.

When examined using SEM, the open cell foams from dispersions 3A through D exhibit cell size gradient from small cells on the outer surface to larger cells in the interior of a foam sheet sample. The character of the foam cell size for all foams is relatively similar, having about 70% to 80% of the cells of size less than 50 microns, about 10-15 % of cell size between 50 and 100 microns and about 10% greater than 100 microns cell size. Foam density for the 4 foam samples vary from 73 g/L for Sample 3A, 97 g/L for Sample 3B, 44 g/L for Sample 3C and 56 g/L for Sample 3D.

Example 4 - Additional Foam Preparations & Testing

In the fashion of Example 3, several different dispersions are prepared, frothed and dried to durable foam. The formulations in the dispersion and froth blends are shown below in Table 3.

TABLE - 3**Polymer Foam Characteristics**

(active component percentages, based on Polymer weight)

<u>Dispersion Designation</u>	<u>Polymer</u>	<u>Dispersant & amount</u>	<u>Frothing Surfactant</u>	<u>Methocel E4M amt.</u>	<u>Wicking Ht. (cm)</u>
3.1	1A	Unicid 425 6%	Stanfax 318 1.7%	0.4%	0
3.2	1B	Unicid 350 6%	Steol CS-130 2%	0.1%	1.4
3.3	1C	Unicid 350 2%	Na Lauryl sulfate 1.5%	0.3%	1.0
3.4	1E	Unicid 425 6%	Steol CS-130 1%	0.2	0
3.5	1J	Unicid 350 6%	Stanfax 318 3.3%	- none -	0.5
3.6	1D	Unicid 350 6%	Steol CS-130 1%	0.2	0
3.7	1D	Erucic acid 3%	Steol CS-130 1%	0.2	3.0
3.8	1D	Erucic acid 3%	Stanfax 318 1%	0.3	1.5
3.9	1D	Oleic acid 6%	- none - (Oleic acid (serves dual purpose)	0.2	2.1
3.10	1D	Oleic acid 3%	(ditto)	0.2	----

Wicking Height Testing

Wicking testing of foams prepared as described above is conducted in the fashion described earlier with 0.9% saline solution. The results suggest that highly hydrophobic dispersants such as the Unicid dispersants render a resulting foam relatively hydrophobic and therefore do not wick aqueous fluid well, but are useful for absorbing hydrophobic fluids (oil spill clean-up, etc.). As Table 3 suggests, aqueous wicking improves when less of a more hydrophobic dispersant is used.

Example 5 - Filler Additive

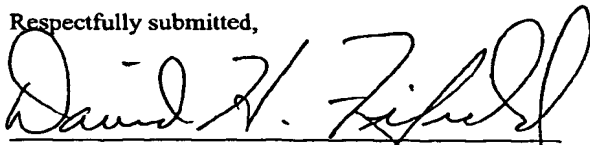
A sample of foam is prepared in the manner described for Foam 3.7 in Example 4, above, except that about 14% of a cotton fiber is added to the blending step before the dispersion is frothed and dried to prepare the Foam of the invention. The resultant Foam is very uniform, has excellent flexibility and softness and exhibits good absorbency and wicking capabilities and a lower elastic character.

PROVISIONAL APPLICATION COVER SHEET

This is a request for filing a PROVISIONAL APPLICATION under 35 CFR 1.53 (c).

Docket Number		63213	Type a plus sign (+) inside this box	+
INVENTOR(s)/APPLICANT(s)				
LAST NAME	FIRST NAME	MIDDLE INITIAL	RESIDENCE (CITY AND EITHER STATE OR FOREIGN COUNTRY)	
Strandburg VanSumeren Wu	Gary Mark Shaofu	M. W.	Mt. Pleasant, Michigan Midland, Michigan Sugar Land, Texas	
<input type="checkbox"/> Additional inventors are being named on separately numbered sheets attached hereto				
TITLE OF THE INVENTION (280 characters max)				
FROTHS AND DURABLE FOAMS OF DISPERSED OLEFIN POLYMERS AND ARTICLES PREPARED FROM SAME				
CORRESPONDENCE ADDRESS				
THE DOW CHEMICAL COMPANY Intellectual Property P. O. Box 1967 Midland, MI 48641-1967 U.S.A.			Telephone: 989-636-2540 Fax: 989-636-3237	
ENCLOSED APPLICATION PARTS (check all that apply)				
<input checked="" type="checkbox"/> Specification		Number of Pages	19	<input type="checkbox"/> Small Entity Statement
<input type="checkbox"/> Drawing(s)		Number of Sheets	_____	<input type="checkbox"/> Other (specify)
METHOD OF PAYMENT (check one)				
<input type="checkbox"/> A check or money order is enclosed to cover the Provisional filing fees			PROVISIONAL FILING FEE AMOUNT	\$160.00
<input checked="" type="checkbox"/> The Commissioner is hereby authorized to charge filing fees and credit Deposit Account Number: No. 04-1512				
The invention was made by an agency of the United States Government or under a contract with an agency of the United States Government				
<input checked="" type="checkbox"/> No				
<input type="checkbox"/> Yes, the name of the U.S. Government agency and the Government contract number are:				

Respectfully submitted,


David H. Fifield
Registration No.: 26,729
Phone No.: (979) 238-4136

DHF/alh

EXPRESS MAIL MAILING NO.: EV318535565 US
DATE OF DEPOSIT: August 25, 2003

PROVISIONAL APPLICATION FILING ONLY

Document made available under the Patent Cooperation Treaty (PCT)

International application number: PCT/US04/027593

International filing date: 25 August 2004 (25.08.2004)

Document type: Certified copy of priority document

Document details: Country/Office: US
Number: 60/497,527
Filing date: 25 August 2003 (25.08.2003)

Date of receipt at the International Bureau: 02 February 2005 (02.02.2005)

Remark: Priority document submitted or transmitted to the International Bureau in compliance with Rule 17.1(a) or (b)



World Intellectual Property Organization (WIPO) - Geneva, Switzerland
Organisation Mondiale de la Propriété Intellectuelle (OMPI) - Genève, Suisse